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June 21, 2001

BOX PCT

Commissioner for Patents
Washington, D.C. 20231

PCT/BE99/00084
-filed July 2, 1999

Re: Application of Alain VANDERGHEYNST, Jean VAN VLIET and Eduard PELCKMANS
PROCESS FOR MANUFACTURING $(U,Pu)O_2$ MIXED OXIDE NUCLEAR FUEL PELLETS FROM NON-FREE-FLOWING UO_2 POWDER
Our Ref: Q64867

Dear Sir:

The following documents and fees are submitted herewith in connection with the above application for the purpose of entering the National stage under 35 U.S.C. § 371 and in accordance with Chapter II of the Patent Cooperation Treaty:

- ☑ an executed Declaration and Power of Attorney.
- ☑ an English translation of the International Application.
- ☑ 3 sheets of drawings (Figs. 1-3).
- ☐ an English translation of Article 19 claim amendments.
- ☐ an English translation of Article 34 amendments (annexes to the IPER).
- ☑ an executed Assignment and PTO 1595 form.
- ☐ a Form PTO-1449 listing the ISR references, and a complete copy of each reference.
- ☑ a Preliminary Amendment

It is assumed that the International Search Report, will be supplied directly by the International Bureau, but if further copies are needed, the undersigned can easily provide them upon request.

Please see the attached PRELIMINARY AMENDMENT before calculating the filing fee.

The Government filing fee is calculated as follows:

Total claims	11	-	20	=		x	$18.00	= $.00
Independent claims	1	-	3	=		x	$80.00	= $.00
Base Fee								$860.00
TOTAL FILING FEE								$860.00
Recordation of Assignment								$ 40.00
TOTAL FEE								$900.00

Checks for the statutory filing fee of $860.00 and Assignment recordation fee of $40.00 are attached. You are also directed and authorized to charge or credit any difference or overpayment to Deposit Account No. 19-4880. The Commissioner is hereby authorized to charge any fees under 37 C.F.R. §§ 1.16, 1.17 and 1.492 which may be required during the entire pendency of the application to Deposit Account No. 19-4880. A duplicate copy of this transmittal letter is attached.

There is no claim to priority.

Respectfully submitted,

John H. Mion
Registration No. 18,879

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PATENT APPLICATION
Q-64867

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Alain DERGHEYNST et al

PCT/BE89/00084
filed July 2, 1999

Appln. No. (NOT YET KNOWN)

Confirmation No. (NOT YET KNOWN)

Filed: June 21, 2001

For: PROCESS FOR MANUFACTURING (U,Pu)O_2 MIXED OXIDE NUCLEAR FUEL PELLETS FROM NON-FREE-

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, D.C. 20231

Sir:

Preliminary to examination of the above-identified Application, please make the following amendments:

IN THE CLAIMS:

The claims are amended as follows:

5. (Amended) The process as claimed in claim 1, characterized in that it furthermore comprises particle size selection by sieving (32) of the granulated UO2 before it is used.

8. (Amended) The process as claimed in claim 1, characterized in that, for said granulation of the non-free-flowing UO2, a lubricant is added to it.

9. (Amended) The process as claimed in claim 1, characterized in that, for said granulation of the non-free-flowing UO2, a binder is added to it.

10. (Amended) The process as claimed in claim 1, characterized in that the sintering (7) of the fuel pellets in an atmosphere of argon and hydrogen is carried out at a temperature between 1600 and 1760°C, the argon possibly being replaced with nitrogen.

11. (Amended) The process as claimed in claim 1, characterized in that, during the sintering (7), the oxygen partial pressure is adjusted, preferably by adjusting the H2/H2O ratio in a flushing gas, in order to improve the interdiffusion of the PuO2 and UO2 oxides.

REMARKS

The above amendments have been made to eliminate all multiple dependent claims (both proper and improper), thereby both ensuring examination of all claims on the merits in the first Office Action and eliminating the need for a multiple dependent claim fee.

Respectfully submitted,

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June 21, 2001

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Registration No. 18,879

APPENDIX

VERSION WITH MARKING TO SHOW CHANGES MADE

IN THE CLAIMS:

The claims are amended as follows:

5. (Amended) The process as claimed in ~~any one of claims 1 to 4~~claim 1, characterized in that it furthermore comprises particle size selection by sieving (32) of the granulated UO2 before it is used.

8. (Amended) The process as claimed in ~~any one of claims 1 to 7~~claim 1, characterized in that, for said granulation of the non-free-flowing UO2, a lubricant is added to it.

9. (Amended) The process as claimed in ~~any one of claims 1 to 8~~claim 1, characterized in that, for said granulation of the non-free-flowing UO2, a binder is added to it.

10. (Amended) The process as claimed in ~~any one of claims 1 to 9~~claim 1, characterized in that the sintering (7) of the fuel pellets in an atmosphere of argon and hydrogen is carried out at a temperature between 1600 and 1760°C, the argon possibly being replaced with nitrogen.

11. (Amended) The process as claimed in ~~any one of claims 1 to 10~~claim 1, characterized in that, during the sintering (7), the oxygen partial pressure is adjusted, preferably by adjusting the H2/H2O ratio in ~~the~~ a flushing gas, in order to improve the interdiffusion of the PuO2 and UO2 oxides.

D129788

Process for manufacturing $(U,Pu)O_2$ mixed oxide nuclear fuel pellets from non-free-flowing UO_2 powder

The present invention relates to a process for manufacturing a $(U,Pu)O_2$ mixed powder from non-free-flowing UO_2 powders.

The manufacture of fuel for light-water reactors, based on uranium and plutonium oxides, generally called MOX fuel, has been the subject of various developments associated with the desire to to recycl plutonium recovered during spent fuel reprocessing.

The manufacture and irradiation of MOX fuel in light-water reactors are now considered to be a solution for providing reasonnable resistance to the proliferation of plutonium present in a form separated from the fission products, whether this plutonium is either of civilian or military origin.

Several processes for manufacturing MOX fuel have been developed over the last two decades, some of the processes involving the complete milling of the UO_2 and PuO_2 powders in order to provide an intimate blend, while others are limited to milling only a fraction of these powders.

The MIMAS (standing for MIcronization and MASter blend) process, which was developed by the Applicant of the present invention (see figure 1), comprises the micronization, by milling, of only a fraction of the final blend and uses two successive blending operations to achieve isotopic homogenization and to take advantage of the use of free-flowing UO_2 incoming products (especially to ensure that the dies of the presses used for pelletizing are properly filled). Using free-flowing UO_2 powders in the second blending operation and limiting the milling to only the first blending operation simplify the manufacture (for example by dispensing with the operations of precompacting/granulating or spheroidization of the

mixed oxide blend) and have greatly simplified, at the start of industrial implementation , the qualification of MOX fuel by users and the licensing process by the Nuclear Safety Authorities (thanks to the similarity in behavior between this MOX fuel and UO_2 fuel).

Various versions of the MIMAS process have been applied, sometimes under names different from MIMAS, but all characterized by two successive blending operations, the second of which uses free-flowing UO_2.

UO_2 which serves as feed material in the manufacture of enriched-uranium fuel and, in the great majority of cases, in the manufacture of MOX fuel, is obtained by the conversion of uranium hexafluoride. There are industrial conversion processes which produce free-flowing UO_2 powder. This is especially the case with two industrial conversion processes using a wet route, known in the art by the respective names "AUC", coming from the intermediate product (Ammonium Uranyl Carbonate), and "TU2", coming from the uranium transformation unit in which the conversion is carried out. One of the drawbacks of these wet conversion processes is the production of a large amount of liquid effluents which have to be treated before discharge. The wet conversion processes, some of which do not produce free-flowing UO_2, are gradually being replaced with dry processes which allow the gaseous effluents to be recycled but which generally produce non-free-flowing UO_2 powder.

For the purpose of diversifying the sources of UO_2 powder for manufacturing MOX fuel by MIMAS-type processes, it is therefore useful to be able to employ non-free-flowing UO_2 powders.

Non-free-flowing UO_2 powder conditioning processes, for transforming it into free-flowing UO_2 granules, and therefore having properties suitable for feeding a pelletizing press, are known. Various mechanical granulation processes, such as precompaction-granulation or agglomeration-

spheroidization, have been developed and are used on an industrial scale in UO_2 fuel manufacturing plants.

Experience has shown that these granulation processes produce granules of insufficient mechanical strength for correct implementation of the second blending operation which characterizes the MIMAS processes and similar processes. Under the optimum operation of the second blender, the granules are damaged and the flowability of the secondary blend is impaired: the fuel pellets which result therefrom suffer from major defects (excessive variability in the physical properties of the product, local differential shrinkage defects, etc.). Alternatively, if the method of operating the second blender is modified so as to achieve gentle mixing of the powders to be blended, or if the apparatus used for the second blending is modified for the same purpose, the uniformity of distribution of the plutonium within the fuel may be impaired and the MOX pellets thus produced no longer meet the maximum plutonium content variability criteria.

To avoid the abovementioned drawbacks, the process for manufacturing MOX fuel from non-free-flowing UO_2 powder, which is the subject matter of the invention, comprises a mechanical granulation treatment of the non-free-flowing UO_2 powder, which does not modify the chemical properties (such as a stoichiometry) and morphological properties (such as the particle size) of the UO_2 powder, but which does nevertheless ensure the mechanical strength and flowability that are required to successfully carry out the second blending operation and the pelletizing operation, respectively.

The invention thus obviates the need to supply the MIMAS-type processes with free-flowing UO_2 powders as feed materials.

According to one advantageous method of implementing the invention, non-free-flowing UO_2 powder is used, one part of which is used, as it is, for

incorporation in the first blend and one part of which undergoes a granulation treatment before being incorporated into the second blend.

In a variant, as a nonlimiting example, said granulation treatment may also be applied to the non-free-flowing UO_2 fraction fed in the first blend

In order to avoid the drawback of the abovementioned lack of mechanical strength of UO_2 granulated by one of the usual conditioning processes, the mechanical treatment according to the invention is carried out either by forcing the non-free-flowing UO_2 powder through a screen or sieve, or by compressing this powder into tablets under a high pressure, as required for obtaining suitable non-friability properties, and then crushing said tablets. When necessary, one or more binders and/or lubricants may be added beforehand to the UO_2 powder.

Further details and features of the invention will become apparent from the claims and from the description of the drawings, which are appended to the present specification and which illustrate, by way of nonlimiting examples, the manufacturing process according to the invention.

Figure 1 shows schematically the steps in the manufacture of mixed oxide fuel according to a known process of the MIMAS type.

Figure 2 shows schematically the steps in the manufacture of mixed oxide fuel according to a process of the invention.

Figure 3 shows schematically variants of the process according to the invention.

In the various figures, the same reference notations denote identical or similar components.

The process of the invention, for the use of non-free-flowing UO_2 powder, comprises basically a process for the manufacture of $(U,Pu)O_2$ mixed oxide fuel pellets, that is to say overall (figure 2):

- dosing and first blending (step 1) of PuO_2 powders and/or UO_2 powders and/or fuel manufacturing scrap;
- micronization (step 2) of this first blend, particularly by milling, and forced sieving (step 3) of its product, for example through a 250 µm screen mesh;
- additional dosing and second blending (step 4) of the first blend thus treated, UO_2 and, where appropriate, fuel manufacturing scrap;
- addition, and blending with the resulting second blend of one or more lubricants and/or poreformers (step 5), the latter step possibly being completely or partly combined with step 4;
- compression (step 6) of the second blend into pellets using pelletizing presses ; and
- sintering (step 7) of the pellets thus formed, preferably in an atmosphere of moistened argon (or nitrogen) and hydrogen.

This mixed oxide fuel pellet manufacturing process may also usually include, for the pellets thus obtained, steps of:

- dry grinding (step 8);
- visual inspection (step 9);
- stacking up to length (step 10);
- loading the pellets into a cladding and welding the latter so as to form a fuel rod (step 11, figure 1);
- pressurizing the rods;
- nondestructive testing/examination of the rods (step 12); and
- assembling of the rods (step 13).

Said process of the invention furthermore includes (figure 2) a prior mechanical granulation treatment of all or part of the nonflowing UO_2 (step 29). This treatment may comprise, for example:

- either (figure 3) steps of compressing the non-free-flowing UO_2 into tablets (step 30) and of

crushing these tablets (step 31) and, where appropriate, of sieving the crushed material (step 32) in order to form free-flowing granules having properties suitable for being incorporated as the basic constituent in the second blending operation (step 4) or, in a variant, in both blending operations (steps 1 and 4), while maintaining the original chemical composition and original particle size of the original UO_2;

- or an agglomeration/precompaction/granulation step by forcing the non-free-flowing UO_2 powder through a screen or sieve (step 29), the amount of additive(s), the mesh size of the screen or sieve and the pressure exerted on the powder being adjusted in order to form granules having the suitable properties described above.

A few nonlimiting parameters of the pellet manufacturing process are given below by way of example:

- batch/campaign operation rather than continuous operation;
- plutonium content of the first blend: 20 to 40% (step 1);
- milling (step 4) in 60 kg batches for a minimum effective time of 5 hours;
- use of non-free-flowing UO_2 powders coming from a wet conversion (for example, ex-ADU or ammonium diuranate powder) or from a dry conversion (said conversions being known to those skilled in the art);
- addition of 0.2 to 0.5% of zinc stearate and 0 to 1% of an AZB pore former (known to those skilled in the art);
- pelletizing compression (step 6) at a pressure between 400 and 700 MPa;
- sintering (step 7) for at least 4 hours at a temperature between 1600 and 1760°C, in an

argon atmosphere containing 5% hydrogen, with an H_2/H_2O ratio of 10 to 30; and

- dry centerless grinding (step 8).

By way of nonlimiting example, the compression step (step 30) may be carried out at a pressure of between 50 and 200 MPa, this being tailored according to the characteristics of the non-free-flowing powder. These pressures are therefore higher than the granulation pressures (4 to 10 MPa) generally used in UO_2 nuclear fuel manufacturing plants. Some binder and/or lubricant, both well known to those skilled in the art, may be incorporated into the non-free-flowing UO_2 powder before compression: by way of nonlimiting example, the compression may thus be carried out at a pressure of between 40 and 100 MPa.

Also by way of nonlimiting example, the aforementioned tablets may be crushed in one or more jaw crushes or roll mills of 200-250 μm aperture. This crushing may be followed by sieving if the crusher lets through, or runs the risk of letting through, granules having a size greater than 250 μm. The fines possibly resulting from the crushing may usefully be incorporated as raw material into the first blending operation (step 1).

By way of yet another nonlimiting example, the operation of forcing the powder through a sieve (step 29) may be carried out in a machine of the kind used in MIMAS-type processes (step 3) to fill the first blend (after the micronization of step 2) before the second blending (step 4). Such machines, which combine agglomeration/precompaction upstream of the sieve and control of the maximum granule size by passing the powder through this same sieve, may produce granules of the desired characteristics directly.

Experience has shown the Applicant that a non-free-flowing powder treated according to the process forming the subject matter of the invention can be used in existing MOX manufacturing plants, by adjusting the parameters of this second blending operation (step 4),

the pelletizing (step 6) and the sintering (step 7), within the adjustment limits routinely used to optimize the manufacturing process according to the characteristics of the various free-flowing UO_2 powders used for MOX fuel manufacture.

The process of the invention therefore makes it possible to extend the range of UO_2 powders which can be used to manufacture MOX fuel, without loosing the benefit of the similarity between the MOX fuel produced according to the invention and the UO_2 fuel manufactured on an industrial scale by the processes known hitherto, starting from the same non-free-flowing UO_2 powder.

It should be understood that the present invention is in no way limited to the methods of implementation described above and that many modifications may be made thereto without departing from the scope of the claims given hereafter.

The non-free-flowing UO_2 conditioning process may especially be applied to UO_2 coming from a conversion other than the conversion of uranium hexafluoride into UO_2.

Claims

1. A process for manufacturing $(U,Pu)O_2$ mixed oxide nuclear fuel pellets,

- comprising:

* dosing and first blending (1) of PuO_2 and/or UO_2 powders and/or fuel manufacturing scrap;
* micronization (2) and forced sieving (3) of this first blend;
* additional dosing and second blending (4) of the first blend thus treated, UO_2 and possibly scrap;
* addition and blending of lubricants and/or poreformers (5), separately or in combination with the second blending step (4);
* pelletizing (6) of the second blend; and
* sintering (7) of the pellets thus formed; and

- furthermore comprising, for at least one portion of the UO_2 powders:

* selection of non-free-flowing UO_2; and
* mechanical granulation treatment (29) of the UO_2 so as to make it free-flowing, before the UO_2 is used as granules in at least said second blending operation.

2. The process as claimed in claim 1, characterized in that it comprises, for said granulation treatment:

* compression (30) of the nonflowing UO_2 into tablets at a pressure greater than that used for the usual UO_2 granulation;
* crushing (31) of the tablets obtained, until a flowing crushed material has been formed; and
* use of at least one portion of this flowing crushed material for said second blending operation (4).

3. The process as claimed in claim 2, characterized in that the compression (30) is carried out at a pressure of between 40 and 200 MPa.

4. The process as claimed in claim 2, characterized in that a jaw crusher or a roll mill is used for the crushing step (31).

5. The process as claimed in any one of claims 1 to 4, characterized in that it furthermore comprises particle size selection by sieving (32) of the granulated UO_2 before it is used.

6. The process as claimed in claim 5, characterized in that the granulated UO_2 is separated, by the sieving (32), into at least two fractions of different particle sizes, the finest fraction possibly being introduced into the aforementioned first blending operation (1) whereas the other fraction is incorporated into the second blending operation (4).

7. The process as claimed in claim 1, characterized in that it comprises, in order to carry out said granulation of the non-free-flowing UO_2, an operation to force the latter through a screen or sieve, the amount of additive(s), the mesh size of the screen or sieve and the pressure exerted on the powder all being adjusted so as to form granules having the appropriate properties.

8. The process as claimed in any one of claims 1 to 7, characterized in that, for said granulation of the non-free-flowing UO_2, a lubricant is added to it.

9. The process as claimed in any one of claims 1 to 8, characterized in that, for said granulation of the non-free-flowing UO_2, a binder is added to it.

10. The process as claimed in any one of claims 1 to 9, characterized in that the sintering (7) of the fuel pellets in an atmosphere of argon and hydrogen is carried out at a temperature between 1600 and 1760°C, the argon possibly being replaced with nitrogen.

11. The process as claimed in any one of claims 1 to 10, characterized in that, during the sintering (7), the oxygen partial pressure is adjusted, preferably by adjusting the H_2/H_2O ratio in the flushing gas, in order to improve the interdiffusion of the PuO_2 and UO_2 oxides.

09/868859

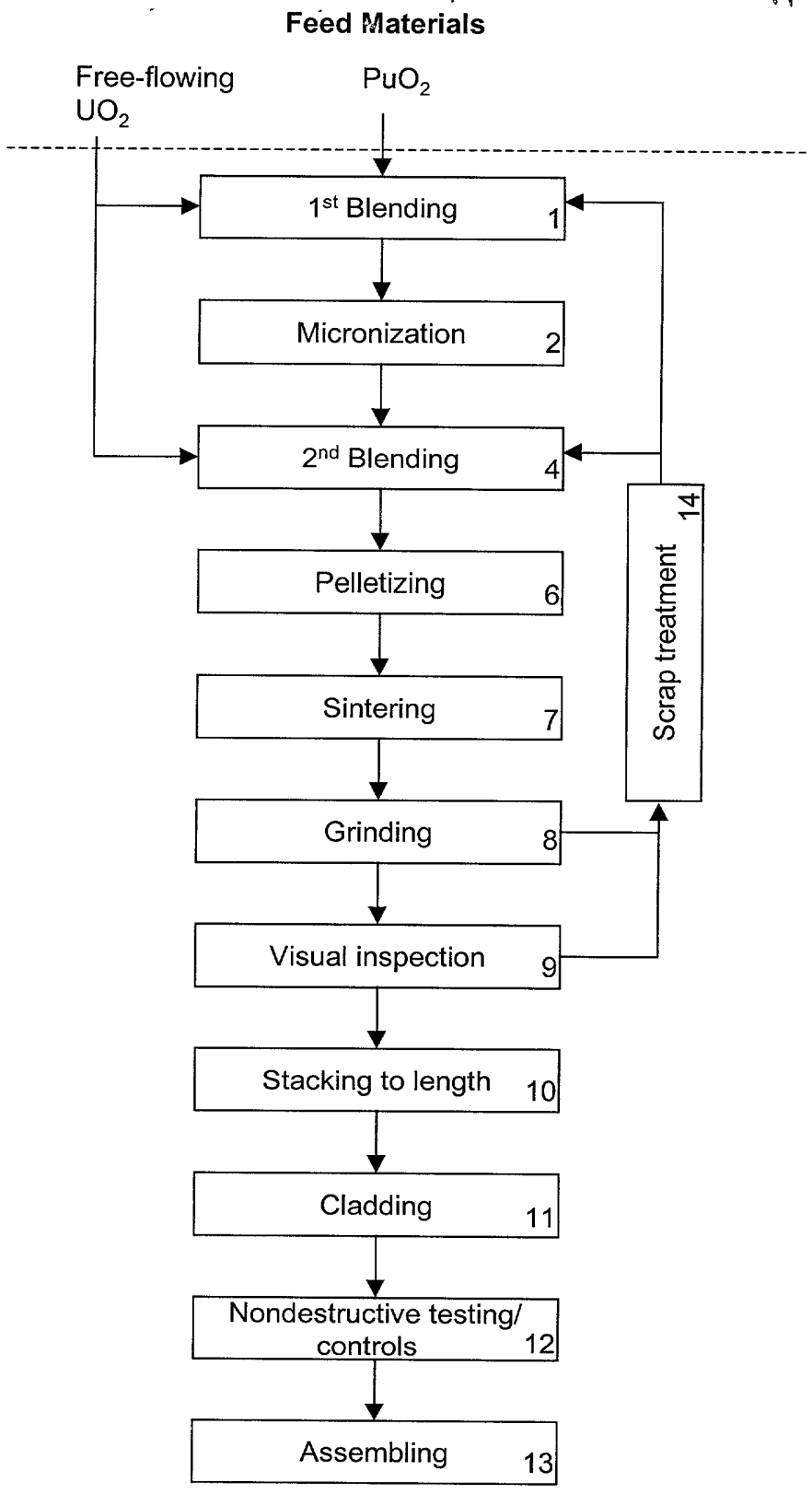

Figure 1

Mixed oxide fuel manufacturing process

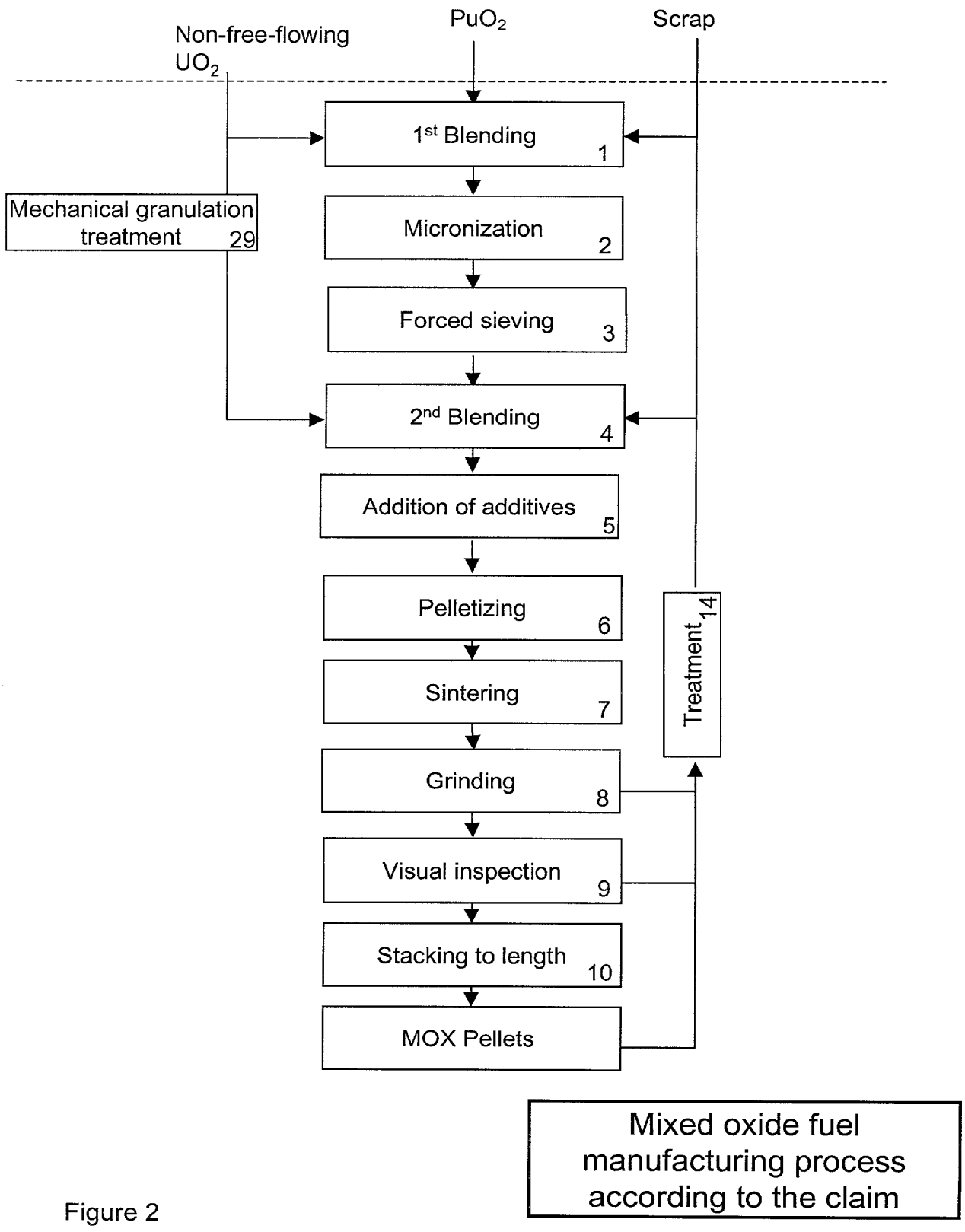
Feed Materials
Non-free-flowing UO_2
PuO_2
Scrap
1st Blending 1
Micronization 2
Forced sieving 3
2nd Blending 4
Addition of additives 5
Pelletizing 6
Sintering 7
Grinding 8
Visual inspection 9
Stacking to length 10
MOX Pellets
Mechanical granulation treatment 29
Treatment 14
Mixed oxide fuel manufacturing process according to the claim

Figure 2

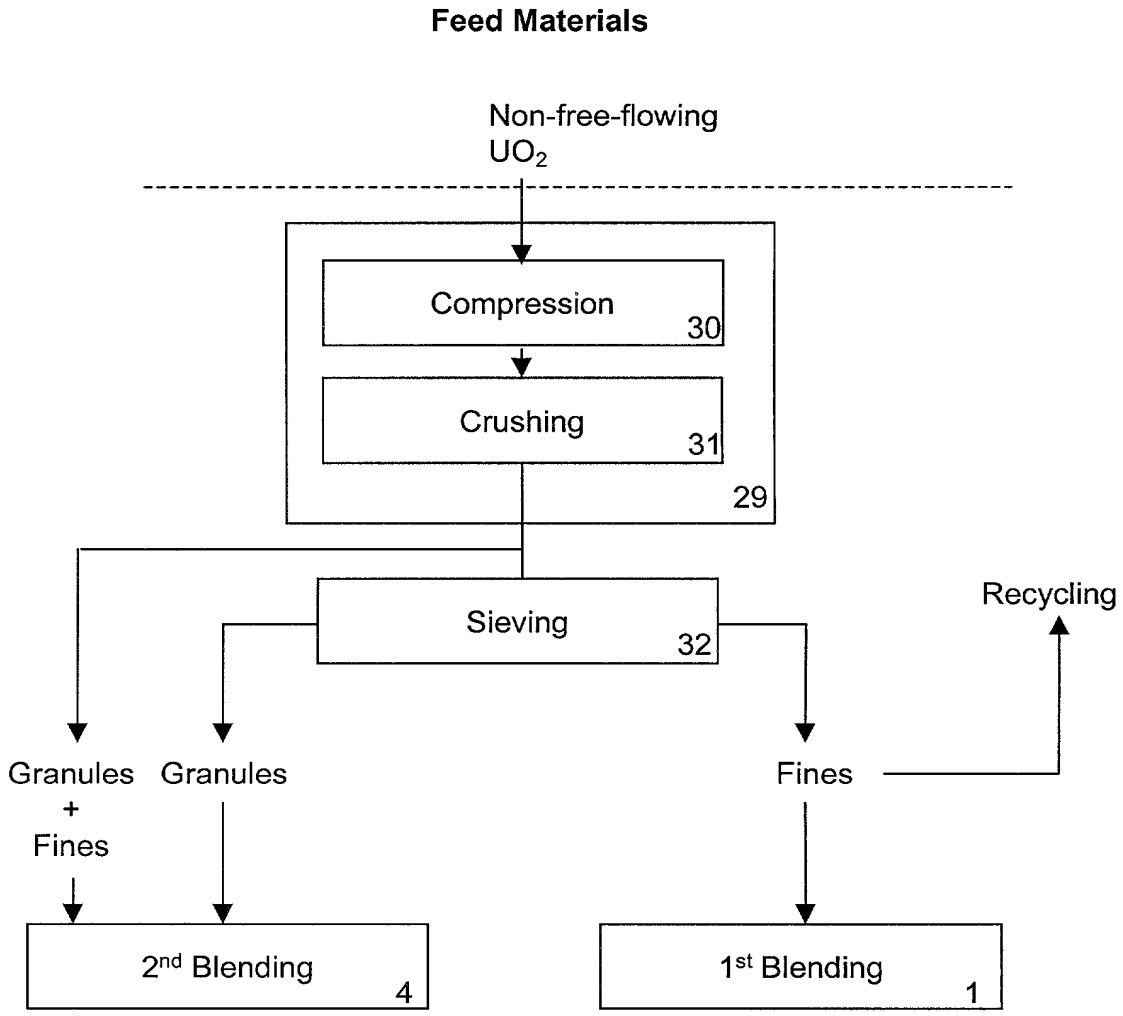

Figure 3

Variant of the mechanical granulation treatment of non-flowing UO_2 powders

SOLE/JOINT

DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that my residence, post office address and citizenship are as stated below next to my name: that I verily believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter claimed and for which a patent is sought in the application entitled:

Process for manufacturing $(U,Pu)O_2$ mixed oxide nuclear fuel pellets from non-free-flowing UO_2 powder

which application is:

☐ the attached application
(for original application)

☑ Application No. PCT/BE99/00084
filed July 2, 1999, and amended on

(for declaration not accompanying application)

that I have reviewed and understand the contents of the specification of the above-identified application, including the claims, as amended by any amendment referred to above; that I acknowledge my duty to disclose information of which I am aware and which is material to the patentability of this application as defined in 37 C.F.R. 1.56, that I hereby claim priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, §119(e) of any United States provisional application(s), or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate or of any PCT International application having a filing date before that of the application on which priority is claimed:

Application Number	Country	Filing Date	Priority Claimed Yes	Priority Claimed No
			☐	☐

I hereby claim the benefit under 35 United States Code §120 of any United States application(s), or §365(c) f any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in a listed prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge my duty to disclose any information material to the patentability of this application as defined in 37 C.F.R. 1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

Application No.	Filing Date	Status

I hereby appoint John H. Mion, Reg. No. 18,879; Thomas J. Macpeak, Reg. No. 19,292; Robert J. Seas, Jr., Reg. No. 21,092; Darryl Mexic, Reg. No. 23,063; Robert V. Sloan, Reg. No. 22,775; Peter D. Olexy, Reg. No. 24,513; J. Frank Osha, Reg. No. 24,625; Waddell A. Biggart, Reg. No. 24,861; Louis Gubinsky, Reg. No. 24,835; Neil B. Siegel, Reg. No. 25,200; David J. Cushing, Reg. No. 28,703; John R. Inge, Reg. No. 26,916; Joseph J. Ruch, Jr., Reg. No. 26,577; Sheldon L. Landsman, Reg. No. 25,430; Richard C. Turner, Reg. No. 29,710; Howard L. Bernstein, Reg. No. 25,665; Alan J. Kasper, Reg. No. 25,426; Kenneth J. Burchfiel, Reg. No. 31,333; Gordon Kit, Reg. No. 30,764; Susan J. Mack, Reg. No. 30,951; Frank L. Bernstein, Reg. No. 31,484; Mark Boland, Reg. No. 32,197; William H. Mandir, Reg. No. 32,156; Brian W. Hannon, Reg. No. 32,778; Abraham J. Rosner, Reg. No. 33,276; Bruce E. Kramer, Reg. No. 33,725; Paul F. Neils, Reg. No. 33,102; Brett S. Sylvester, Reg. No. 32,765; Robert M. Masters, Reg. No. 35,603, George F. Lehnigk, Reg. No. 36,359, John T. Callahan, Reg. No. 32,607 and Steven M. Gruskin, Reg. No. 36,818, my attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith, and request that all correspondence about the application be addressed to SUGHRUE, MION, ZINN, MACPEAK & SEAS, PLLC, 2100 Pennsylvania Avenue, N.W., Washington, D.C. 20037-3213.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

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Citizenship Belgian

Date Fourth Inventor
First Name Middle Initial Last Name

Residence Signature
City State/Country

Post Office Address:

Citizenship

Date Fifth Inventor
First Name Middle Initial Last Name

Residence Signature
City State/Country

Post Office Address:

Citizenship

Date Sixth Inventor
First Name Middle Initial Last Name

Residence Signature
City State/Country

Post Office Address:

Citizenship